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MELLON INSTITUTE

Report No. 13

MONTHLY SCIENTIFIC PROGRESS REPORT

For the month of July 1961

STRESS CORROSION OF HIGH STRENGTH STEELS AND ALLOYS; ARTIFICIAL ENVIRONMENT

Research Project No. 389-1

Sponsored by U. S. Army Ordnance, Frankford Arsenal Mr. H. Rosentha', Contract Monitor

Contract No. DA-36-034-ORD-3277RD

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The publication of this report does not necessarily constitute approval by the Army of the findings or conclusions contained therein.

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L INTRODUCTION

The project work described herein represents a portion of a grant made available by the Army to promote a general scientific advancement in the area of case materials for missiles. This specific project is concerned with the synthetic environment stress corrosion testing of specified high strength steels and alloys. The research objectives of the project were presented in the July Monthly Scientific Report, Report No. 1.

Natural environmental tests on high strength steels and alloys are being conducted by Aerojet General Corporation, with actual production environments and rocket propellant environments being utilized. By prior mutual agreement, the same steel and alloy sheet material will be used for both projects and possible heat treatment variations will be circumvented by exchanging heat treated material whenever possible.

A number of drawings and schematic diagrams of apparatus and test methods pertinent to the project have been presented in prior reports. In addition, summarized surveys of applicable industrial and military literature have also been presented previously (August and September rex-

This report presents further information on continuing U-bend and bent beam tests for the assigned alloys, and for Rocoloy 270, 4137 Co. and Ardeform 301 missile steels.

II. EXPERIMENTAL PROCEDURES

Test Methods

A discussion of the U-bend and bent beam test methods together with an outline of the synthetic stress corrosion test environments used in performing the research are given in the July, 1960, Monthly Scientific Report, Report No. 1.

All recently-exposed (and future) samples being subjected to stress corrosion testing are being weighted prior to exposure to the test solutions to provide, whenever possible, general corrosion information.

A TRATATUS

Schematic drawings of bent beam sample holders, the U-bend test and holders, a sample bending device for bent beam specimens, and a stress corrosion test tank were presented in Report Nos. 1 and 3.

Polyethylens containers are presently being used for the stress corrosion exposure of U-bend specimens to the various synthetic environments. Each container will adequately hold six U-bend specimens. The use of these containers will supplement the samples presently being tested in epoxy-coated tanks.

Construction of shelving and an aeration system (Report No. 6) together with other pertinent items has facilitated (wherever

feasible) the transfer of samples under test from the glass containers to the epoxy-coated test tanks. The completed facility is illustrated in Report No. 7.

Alloy Sample Material

The alloys being used in the study on scress corrosion include:

- 1. Low Alloy: Ladish D6Ac.
- 2. Si-Modified 4300 Series: 300 M.
- 3. Hot-Worked Die Steel: Vascojet 1060.
- 4. Cold-Worked PH Steel: AM 355.
- 5. Heat-Treated PH Steel: PHI5-7 Mo.
- 6. Titarium Alloy: B120VCA.
- 7. Low Alloy-Cobalt Modified: 4137 Co.
- 8. Low Alloy-Cobalt Modified: Rocoloy 270.
- 9. Stretch-Formed 17/7 Stainless Steel: Ardeform 301.

All of the sample material is being tested in sheet form and was procured as such.

Chemical analyses for the above listed alloys and for the comparative heats are given in their entirety in Table I. Physical properties and heat treating surveys for the alloys were presented in Report No. 12.

The status of the alloy sample material is as follows:

- 1. DóAc ready for final machining
- 2. 300M ready for final inachining
- 3. Vascojet 1000 ready for heat treatment
- 4. AM355 under test
- 5. PHI5-7Mo ready for heat treatment
- 6. Bl20VCA being machined
- 7. 4137 Co under test
- 8. Rocoloy 270 ready for final machining
- 9. Ardeform 301 under test

On all of the above listed alloys, the final machining step consists of wet grinding the specimens to thickness ($.051 \pm .001$). After heat treating, all specimen surfaces (except ends) are hand polished with 240-grit emery paper.

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III. EXPERIMENTAL

Sample preparation on all alloys noted in the aforementioned section is continuing.

U-bend stress corrosion testing is continuing for the allogs, including 4137 Co, AM355, PHI5-7Mo, BI20VCA and Ardeform 301.

Bent beam specimens of 4137 Co and AM355 are still under test.

The foregoing specimens under test are discussed in greater detail as follows:

Bent Beam Tests

Bent beam specimens of AM355 (secondary direction) are undergoing stress corrosion testing. These specimens were cold-rolled to their strength level (250, 261, 302 Kpsi) by the vendor. To date there have been no failures, as indicated in Table II. Visual observation shows no indication of general corrosion on the AM355 specimens. It is expected in the near future that the primary direction of the AM355 bent beam specimens will also be under test.

In the cooperative program with U. S. Steel Applied Research

Laboratory, in which bent beam specimens of 4137 Co specimens were

exposed to three natural environments, little change has been noted in

the status of the specimens.

The U. S. Steel Applied Research program utilizes samples of 4137 Co heat treated to three strength levels and then stressed to 75% of their Y.S. The specimens were exposed to two ratural environments: Kure Beach, N.C. (marine) and Monroeville (semi-industrial). Table III gives the cumulative data on the specimens to date. It may be noted that the status of the specimens has not changed over the past few months.

The program on 4137 Co bent beam specimens conducted by this Project utilized the same heat of sample material and the same heat-treating procedures. An industrial environment, Pittsburgh, Pa., was chosen for testing. Specimens of the three Y.S. levels were stressed to 50%. 75% and 90% of their Y.S. The cumulative results on the specimens used by project personnel is given in Table IV. It may be noted that very little change has taken place over the last few months and only scattered failures have been reported.

U-Bend Tests

The U-bend specimens of the high Y.S., primary direction, of AM355 are presented in Table V. Since the specimens are only from one strength level and one direction, no correlation can be made between U-bend and bent beam results for AM355. The remainder of the U-bends for both directions and all strength levels are expected to be under test in the near future.

It can be seen from Table VI that no failures have occurred with the BLCVCA U-bends. These specimens are, however, from the lowest strength level (135 hpsl) and the secondary direction.

The U-bend specimens of the PHI5-7Mo alloy are still undergoing testing. It is shown in Table VII that no failures have occurred to date. The specimens were taken in both the primary and secondary directions and wer, cold-rolled to a strength level of approximately 195 Kpsi by the vendor.

Additional U-bend specimens of PHI5-7Mo and AM355 are being tested in three additional test solutions above and beyond those assigned for this project. These test solutions include:

- a chlorimated non-hydrocarbon (CCl₄);
- 2. a non-chlorinated hydrocarbon (C_5H_{12}); and
- 3. a chlorimated hydrocarbon ($C_2H_2Cl_4$).

Table VIII gives the cumulative results on the aforementioned test.

No recent failures have resulted from the foregoing tests, exclusive of the one early failure of a PHIS-7Mo sample in CCl₂.

Ardeform 301 specimen testing is continuing in the five test solutions, namely: NaCl, NaNO₃, Na₂SO₄, NaPO₅ and Na₂S. The cumulative data on sample No. 1 of the Ardeform 301 material is given in Table IX. The results indicate a susceptibility of the Ardeform 301 in the one molar NaCl test solution. Sample No. 2 of the Ardeform 301

does not show the same degree of susceptibility as shown in Table X.

Monthly Progress Report No. 10 presents the sample preparation information for Samples 1 and 2.

U-bend tests on 4137 Co, a low alloy missile steel, are continuing. It can be seen from Table XI that the specimens in the one molar NaNO₃ and NaPO₃ have failed for all three strength levels. The Na₂S test solution appears to have only a slight effect on the 4137 Co specimens.

The 4137 Co results on the effect of water vapor exposure are presented in Table XII. As the data shows, the "Humid Air" test was the only one with an effect on the specimens. There have been no failures with the specimens tempered at 1100°F in the "Riumid Air."

IV. FUTURE WORK

Sample preparation, heat treatment, metallographic examination, and stress corrosion testing will constitute the main effort of the project.

Photo-micrographs of specimens from the heat treatment survey will be presented together with grain size determinations.

Cumulative test results on bent beam and U-bend tests will be presented as they become available.

Future reports will be presented on a quarterly rather than a monthly basis.

C. J. Owenigra

W. D. Ruble

R: 8-22-61 T: 8-22-61 FELLOW

PESEABLE ASSISTANT

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CHEMICAL ANALYSIES OF STEELS AND ALL OYS

Steel Alloy or	Mample Dealgnarion	b	Min	1		1 1 1 1 1 1 1 1 1 1	3 3	Cr NI Mo	72 22 24 25 25 25 25 25 25 25 25 25 25 25 25 25	SI CF NI Mo AI	>) (30 1	z	11:	;
0000	Pelmary Haat	0, 48	0, 68	0, 007	0,008	0, 22	1.23	0, 58	1.0		ეი 'ი			-	bal.
D6Ac	Comp. 10 #2	0.48	0, 70	0.010	0,00%	0, 19	1.19	79'0	1.08		0,10			~	bal.
300M	Primary Heat	0, 43	0, 66	0, 006	0. UNB	1, 80	0, 84	1.83	0.35	0, 065 0, 06	0.06			-	bnl.
SOOM	Comp. III #1	O. 41	0, 70	0, 007	0,004	1, 50	0. 77	1. 71	0, 17	0,11	0.09			_	bal.
V-1000	Primary Haat	0, 38	97.0	010 '0	0, 008	08 '0	5, 10		3, 15		0, ·18		•	-	bal.
0001-7	Comp. 11 //	0. 38	0, 22	0, 00%	0,007	0,90	8, 19		1.20		0.50			_	bal.
000 T- A	Comp. 111 #4	0, 39	0.27	0,009	0,008	9, 98	5.1.		1.29		0.5			_	bal.
0001-7	Comp. III # 3	0, 42	0, 36	0, 011	0,006	0,96	0,96 4,90		1, 28		0.50			_	but.
A MJ55	Drinney Hoat	o, 13	0.72	0, 018	0.018	0.29	0, 29 15, 60	4. 38	2,71				0.11		bul.
PHB-7M0	Primary Heat	80 °O	0. 54	0.014	0, 008	0,26	0, 26 15, 05	7. 12	91 '7	1.16				_	bul.
MZOVCA	Primary Boat	20 '0					2.11	0,02		3.0	13.7	0.0	0.018	hal.	n 23
MZUVCA	Comp. 111 #1	0,03					10.01	0.07		2,95	13, 30	0.012		bal.	0,68
4137 Cu	Vac. Mult	77. *0	0.77	0,009	0.008	0.90	1.14	0,12	0,26	20.0	0.16	1.10		-	bal.
H270	Air Muli	0.48	F. 9	0,008	0, 004	1, 38	0.95	1,18	3, 45	0.12	0.26	v. 9k		-	bal.
R270	Vac. Molt	91.0	0.96	000.0	0.006	1,08	1.65	1.29	0.84	0.038 0.20	0.20	1, 02			bal.
Arde, 301	Cold Wkd 8, 8.	0, 06	0, 06 1, 52	0, 030	0.016	0.49	0, 49 17, 02	7.50						_	bal.

0 . O

11.

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CUMULATIVE AM355 STRESS CORROSION BRITT REAM TESTS

Secondary (tilghent Y. B.) Diruction.

Tout Solution	Approx. Tunello Strong on Sampleto	Number of Samples	Falluron to Oatu	Pailuron Average Time to Pallare Time to Into	Rango, dayo
Naci, 1 M	180 kps f	9	none to 153 days	ž :	1
	198	•	none to 137 days	1 1	: :
	226	9	none to 150 days	I I	:
Nazistoja 1 M	180	3	none to 153 days	:	3 t
•	201	•	none to 137 days	1 1	1
	927	૭	none to 150 days	2 2	:
Nano, 1 M	091	9	none to 153 days	ŧ	: 1
2	961	•	none to 137 days		:
	220	ઝ	none to 150 days	1 1	1
NAPO . 1 M	081	3	none to 153 days	1	1
•	11.75	ઝ	none to 137 days	1	•
	223	ઝ	none to 150 days	# #	1
Na. 5, 1 M	180	9	nonu to 153 days	£ 1	1
ı	195	•	none to 137 days	1 1	1
	225	•	none to 150 duys	1 2	1 8

a All anniples atronsed in holder to 75% of Y.S.

111 3116 VI

CUMULATIVE 4137 Co STRESS CORROSION BENT BEAM TEST'S*

Primary (Lowant Y.S.) Direction

Austonitizing Comporatoro	Tomportuga Tomporaturo	Approximato Tonsilo Stross ^{mb}	Numbur of Samples	Fallures to Date	Avarage Time to Fallure, days	Fallure Time
Marino Exposu	Marine Exposure (Kure Boach, North Carolina)	North Carolina)				
1,000t	980°I° 780 1100	190 kp#1 160 130	ଅକ୍ତ	3 5 None to date	æ <u>.</u>	3 to 16 6 to 18
		* 1 = 01	tests bogun 10-26-60	03		
Somi-Induscria	d Exposure (Mon	Somi-Industrial Exposure (Mouroeville, Pennsylvania)	•			
3,00 . T	550°F° 750 1100	190 kps i 160 130	ស ឃ ឃ	5 8 None to date	9 8 1 9 2 1	2 to 30 18 to 45
		tosts	tuntu bugun 10-26-60	ε.		

* Cooperative touting progrem with U. S. Stool Applied Renoused Lab., Monroeville, Pa.

VI 81.181 AT

CUMULATIVE 4137 C. SPRESS CORROSION BENT BEAM TESTS

Prinnry (Lowest Y.B.) Direction

ttelbitrgh, Penneylvania) 50 50 130 kps 1 16 50 75 75 175 76 76 76 77 79 79 70 70 70 70 70 70 70 70 70 70 70 70 70	Auntonitizing Pamporitira	กลหายสอบเมอส, สีเปลาอยู่เบอส,	% of Y.B.	of Y.S. Approximate Number of Fallaces Average Time to 1	Number	Antibor of Pathieun	Avor	ago Timo to	rallaro Timo
550°F 50 130 kpu1 6 1 to 151 days 750 50 115 6 none to 151 days 1100 50 195 6 6 47.2 150 75 175 6 1 to 151 days 150 75 179 6 1 to 151 days 150 75 140 6 1000 to 151 days 150 200 6 3 to 151 days 1100 90 170 6 none to 151 days	Suvere Industrie	ni iskpoure (Pig	tuburgh, Pan	nnylvania)					William Talling
850 75 175 6 1 47.2 750 75 175 6 1 to 151 days 1300 75 140 6 140 150 90 230 6 6 3 47.2 150 90 230 6 3 to 151 days 1100 90 170 6 none to 151 days	1700°E	550 FF 750 1100	3 5 5 3 5 5	130 kps.1 118 98	၁၁၁	1 to 151 none to 151 none to 151	days days days	1 1 1	: : :
560 90 230 6 6 3 to 151 du; 170 6 none to 151 day	1700	850 750 1100	27. 25.	196 176 140	૦૭૭	6 1 to 151 o	dnye	4 1 1	30 to 65
	1700	360 760 1100	0 6 6 6 6	230 200 170	999	6 3 to 151 c none to 151 c	du;**	33.6	21 to 43

V 21.18.A

CUMULATIVE AMJES STRESS CORROSION U-BEND TESTS

Primary (Lowest Y.S.) Direction

Tost Solution	Tost Solution Y.S. (?% Offsot) Number of Trast Loves, Kprf Samples	Number of Samples	Fulluros to Duto	Average Time to Fallare, days	Lutturo Timo tange,
NaCJ	226 (nu ruevd)	9	9	3.25	0.5 to 8
NaNO	226 (am rocvd)	ŋ	none to 118 days	1 2	1 2
S ⁷ NN	226 (ny rogyd)	•	none to 116 days	1 1	1 2
Na2504	226 (as rouvd)	9	none to 118 days	1 1	† 1
No PO.	226 (na rugvd)	ŋ	none to 116 days	1	1 1

Note: Outur surfece of U-band specimens strussed bayond the Y.S.

TABLE VI

CUMULATIVE BILO VCA STRESS CORROSION ULBEND TESTS

Secondary (Highaut Y.S.) Direction

Test Solution	Y.S. (.2% Office) P. Tout Lavol, Kput	Number of	Fulluron to Duto	Avaraga filmo to Fallaro, days	Tout Solution Y.S. (. 2% Office) Number of Fullares Average True Range Tout Invel, Kpul Samples to Date to Fuffare days (days)
Nict, 1 M	135 (no raced)	÷	none to 207 days	1 1	:
NaNO3. 1 M	1 ·B (au rucvd)		neno to 207 days	i i	\$ \$
NaPO ₃ . 1 M	135 (an rocvd)	7	nona to 207 dayn	1	1 1

Notes Outer surface of U-bond sumples is stressed beyond the Y.S.

LABLE VII

CUMULATIVE PHB-7 NG STRESS CORROSION U-BEND TESTS

Tent Solution Diesetton	Of Specimen	Number of	Palluron to Date	tent Solution Diraction Namber of Palluren Avorage Line Fallure Thing Kanga of Spreamon Samplen to Pallure, dayn (dayn)	Faffure Time Kam (days)
Naca	Primary Sucondary	૭ ૩	nong to 124 days		8 8 2 4
NANO	Primary Secondary	. •	none to 124 days none to 124 days	: :	1 1
S''S	Primary Steondary	9 9	none to 344 days none to 124 days	: :	: a : 1
OS. VN	Primary Secondary	£-0	none to 124 days	1 1 1	\$ 2 2 g
Na PO ₃	Primary Sucondary	ss	none to 124 days	1 1 1	3 3

Notes Outor murface of U-bond specimens stressed beyond the Y.S. Primary (townst Y.S.) and Secondary (highest Y.S.).

TABLE VIII

CUMULATIVE PINS-7 MG AND AMJSS STRESS CORROSION U.BEND TESTS

Strung Corrogian Environment	Type of Steel	Sample Direction	Y. S. Kpui	Y. S. Number Kpsi of Samples	Falluros to Date	Average Time to Fallure, days	Fallure Time
Carbon Totrachloride	PHB-7M0	Primary	201		l to 52 days	:	;
້າວວ	PHI:-7Mo	Secondary	812	\$	none to 52 days	3	: 2
(Chlorinated nc.:	AM355	Primary	226	•	none to 52 days	:	1
hydroca rbon)	AM355	Primary	215	9	none to 52 days	3	1
Puntane	PHIS-7Mo	Primary	201	•	none to 52 days	3	:
CSHZ	PHI5-7M0	Sucondary	812	•	none to 52 days	•	
(Non ahlormated	A M 355	Primary	977	•	none to 52 days	2	
hydrocarbon)	A M355	Primary	215	•	none to 52 days	i e	1
Trichlorothane	FH115-7Mo	Primary	201	æ	none to 52 days	3 2	;
CZHZCI	PHI5-7Mo	Secondary	818	•	none to 112 days	1	:
(Chlorinated	A M355	Primary	927	•	none to #2 days	1 1	*
hydrocarbon)	AM355	Primary	215	•	none to 62 days	1	1

Notes Outer surface of U-bend specimens stressed beyond the Y.S.

TABLE IX

CUMULATIVE ARDEFORM 301 STRESS CORROSION U-BEND TESTS

Stream Corrogion Environment	Sample Direction	Tout Surface in Tonsion	Number of Sumples	Falluros to Dato	Average Time to Failure, days	Falluro Timo Rango, (dayo)
Sample No. 1						
NaCI, 1 M	Longitudinal	Cutaide (convex)	•	4	1,25	0. % to 2
	Longitudinal	Inside (concave)	*	2 to 118 days	<u>.</u>	1 2
	Truns.	Outside	•	none to 118 days	:	1
NaNO, , M	Long.	Outeide	•	none to 118 days	;	3 2
•	Long,	Incide	*	none to 118 days	1 2	:
	Trans.	Outside	9	none to Ile days	!	2 2
NA2S, 1 M	Long.	Outsido	•	none to 118 days	;	ŧ :
•	Long.	Inside	•	none to 118 days	:	:
	Trans.	Outside	9	none to 118 days	:	:
N#2504. 1 M	Long.	Outside	*	none to 118 days	;	:
	Long.	Inelde	•	none to 118 days	:	:
	Trum.	Outeide	9	none to 118 days	1 1	:
NaPO3, 1 M	Long.	Outeidn	•	none to 118 days	:	:
	Long.	Inside	•	none to 118 days	•	* • • • • • • • • • • • • • • • • • • •
	Truns	Outside	•	mone to 118 days	1	1 1

Note: Outer surface of U-bend sumples is stressed beyond Y.S.

TABLEX

CUMULATIVE ARDEFORM 301 STRESS CORROSION U-BEND TESTS

Stress Corregion Environment	Sample Direction	Tout Surface	Number of Samples	Fallures to Onto	=	Average Tinic rallure Tinic Range, Frilure, days
Sample No. "						e escuesa soutes e sus soutes se un faise de la companya de la companya de la companya de la companya de la co
NaCl, 1 M	Longitudinal	Outwide (convex)	9	none to 61 days	i	3 1
	Longitudinal	Inside (concave)	7	1 to 61 duys	4 1	;
	Transacrec	Outuido	n	none to 61 days	: 1	3
NaNO3, 1 M	Long.	Outsido	×0	none to 66 days	•	
•	Long.	Insido	*	none to 66 days	:	8 2
	Trans.	Outerde	থ্য	none to 66 days	1	:
NA,S, 1 M	Long.	Outside	*	none to 66 days	2 3	:
ı	Long.	Linido			1	1 3
	T'runs.	Outeldo	æĵ.	none to 60 days	# #	
Na Sout 1 M	Long.	Outside	•	none to 66 days	1 8	1
	Long.	Insido		none to 66 days	:	1 .
	Trans.	Outside	2 0	none to 66 days	:	1 :
Na PO.	Long.	Outside	7	none to 66 days	i	1
•	Long.	Inside	•	none to 66 days	:	1 1
	Trans.	Outside	×	rone to 66 days	: 1	:

Note: Outer surface of U-bond samples is stressed beyond Y.S.

131.E X

CUMULATIVE 4137 Co STRESS CORROSION U-DIGND TESTS

Primary (Lowant Y.S.) Direction

Tout Solution	Youperature	Tomportul	Number of	Furluran to Date	Average Times to Fallare	Failure Time Range,
NaCi, 1 M	.4.004F	550 250	94		٠٠.3	8.60 1.08
		0011	. •	1 to 375 days	1 ±	1 1
NIL, SO, 1 M	1.500°F	989	3	J	٠.0	10 min. to 2, 5 days
		750	ত ত	none to 315 days	1 8 2 9	
N I WONT	1700°18°	30 30 30 30 30 30 30 30 30 30 30 30 30 3	•9	9	2.5	0, 5 to 1, 5
'n		750	· • •	. • • •	33.8	29.5 to 39.5 26.0 to 277.0
M 1 fodns	1700°F	550 750 1100	399	. ૩૩૭	4.5 min.	34 min. to 5 days.
Na.s. 1 M	1700°£	850 750 1100	39 9	2 to 181 days I to 181 days none to 180 days	1 2 2	::.

* One specimen lesting 5 days not awraged,

Note: Outor surface of U-bond samples is atressed beyond the Y.S.

TA 111.F. XII

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CUMULATIVE 4137 Co STRESS CORROSION U-BEND TESTS

Primary (Lowast Y.S.) Direction

Environment	Austonitizing Temporaturo	Tempering Sumber of Temples	Number of Samples	Number of Fullures	Average Time to Fallure, days	Avorage Time Fallure Time Range. o Fallure, days
Dry Air (dossicator)	1.00.L	750	20 7	none to 181 days	: 1 : 1 : 1	1 1 0
Hunid Air (satur, with water vapor)	7,00t1	250 750 1100	ে কক	4, 4 nears to 173 days		4 to 11, 5 2, 5 to 143
Laboratory (exposed directly to lab, environ,	Mecul.	286 780 1100	444	none to 173 days none to 173 days none to 173 days	: : :	: : :

A One specimen lasting 143 days not averaged.

Note: Outax enriace of U-band samples is strussed beyond the Y.S.

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